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Indian Standard

SPECIFICATION FOR 3-BROMOBENZANTHRONE

(*First Revision*)

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Gr 3

May 1985

AMENDMENT NO. 1 MAY 2002
TO
IS 6961 : 1984 SPECIFICATION FOR 3- BROMOBENZANTHRONE

(First Revision)

(*Page* 3, *clause* **0.3**, *Structural Formula*) — Insert the following below the structural formula:

'(CAS No. 81-96-9).'

[*Page* 4, *Table* 1, *Sl No.* (vii)] — Delete.

[*Pages* 9 *and* 10, *Appendix* A, *clause* **A-5**) — Delete.

(PCD 11)

Reprography Unit, BIS, New Delhi, India

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SPECIFICATION FOR 3-BROMOBENZANTHRONE

(First Revision)

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(*Continued on page* 2)

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Indian Standard

SPECIFICATION FOR 3-BROMOBENZANTHRONE

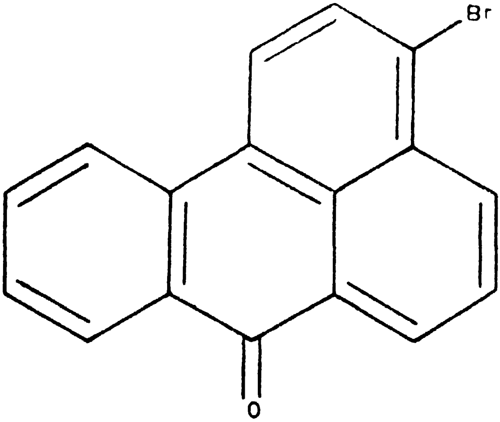
(First Revision)

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 10 December 1984, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1973. Taking cognizance of experience gained in manufacture and also the quality of the material now commercially available in the country, the Sectional Committee decided to revise the standard. In this revision subsidiary constituents namely, benzanthrone and 3, 9-dibromobenzanthrone have been stipulated quantitatively and their chromatographic separation and estimation by spectrophotometer given. The moisture content of the material has also been revised.

0.3 3-Bromobenzanthrone is an important intermediate used in the manufacture of vat dyes. It is represented by the following structural formula:



3-Bromobenzanthrone
(Molecular Mass 309·2)

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the method of sampling and test for 3-bromobenzanthrone, technical.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of yellow powder and shall be free from visible impurities.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR 3-BROMOBENZANTHRONE, TECHNICAL

SL No.	CHARACTERISTIC	REQUIREMENT	METHODS OF TEST (REF TO CL NO. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	3-Bromobenzanthrone content, percent by mass, *Min*	88·0	A-1
ii)	Benzanthrone, percent by mass, *Max*	4·0	
iii)	3, 9-Dibromobenzanthrone, percent by mass, *Max*	4·0	
iv)	Melting point, °C, *Min*	163	A-2
v)	Moisture, percent by mass, *Max*	1·0	A-3
vi)	Sulphated ash, percent by mass, *Max*	1·0	A-4
vii)	Bromine content, percent by mass,	25·5 to 26·5	A-5

3. PACKING AND MARKING

3.1 Packing — The material shall be suitably packed in steel drums lined with polyethylene bags or as agreed to between the purchaser and the supplier.

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*Rules for rounding off numerical values (*revised*).

3.2 Marking — The containers shall be marked with the following:

a) Name of the material;
b) Tare, gross and net mass of the material;
c) Name of the manufacturer and his recognized trade-mark, if any;
d) Batch No; and
e) Date of manufacture.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in **3** of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Test for the determination of 3-bromobenzanthrone, benzanthrone and 3, 9-dibromobenzanthrone shall be conducted on each of the individual samples.

4.2.2 Test for the determination of remaining characteristics, namely, description, melting point, moisture, sulphated ash and bromine content shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirement of 3-bromobenzanthrone, benzanthrone and 3, 9-dibromobenzanthrone if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 *For Composite Sample* — For declaring the conformity of the lot to the requirements of all other characteristics (*see* **4.2.2**) tested on the composite sample, the test results for each of the characteristics shall satisfy the relevant requirements given in **2.1** and Table 1.

---

*Methods of Sampling and tests for dye intermediates.

5. TEST METHODS

5.1 Test shall be carried out according to the methods prescribed in Appendix A, as indicated in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(*Table* 1, *Clause* 5.1)

METHODS OF TEST FOR 3-BROMOBENZANTHRONE

A-1. DETERMINATION OF 3-BROMOBENZANTHRONE, 3, 9-DIBROMOBENZANTHRONE AND BENZANTHRONE

A-1.0 Outline of the Method — The purity of the sample is estimated by employing the chromatographic method. 3-Bromobenzanthrone, 3, 9-dibromobenzanthrone and benzanthrone are separated chromatographically and determined quantitatively by spectrophotometer.

A-1.1 Reagents

A-1.1.1 *Toluene* — Dry for 24 hours over anhydrous calcium chloride filter and distil. Collect the portion boiling at 110°C. The optical density shall not exceed 0·005 at 395, 400 and 385 μm.

A-1.1.2 *Alumina* — Column chromatography grade which is neutral and shall pass through 160-micron IS Sieve. To get material of desired activity treat alumina anhydrous neutral as follows:

> Spread out the alumina evenly on stainless steel tray in a thin layer, add 1 percent of water slowly from a burette covering the whole of the tray as evenly as possible. Mix the alumina by hand initially and then transfer to a kilner jar and mix by rolling mechanically for at least 1 hour. Then retest for activity. If still too active repeat the procedure adding more water in the increment of 0·25 percent.

---

*Specification for water for general laboratory use (*second revision*).

A-1.2 Preparation of Sample

A-1.2.1 Dissolve about 0·5 g of the sample in minimum amount of toluene. Transfer the solution gradually to the chromatographic column. Elute further by toluene. Collect the first band of 3-9-dibromobenzanthrone. Concentrate the solution of 3-9-dibromobenzanthrone by evaporation until crystalization occurs on cooling to room temperature. Filter off the crystals and dry to constant mass in a vacuum oven at 70 to 80°C. To check the purity of material dissolve about 0·01 g in minimum quantity of toluene and pass the solution through a chromatographic column. Set up as described above. Elute as necessary with toluene, collect the bands carefully and determine the optical density at 400 μm.

A-1.2.2 Make up a solution of crystals in solvent. Determine its optical density in the same way.

A-1.2.3 A difference in optical density of more than 0·003 indicates the impurities present in the purified 3-9-dibromobenzanthrone. In that case repeat the chromatographic purification until this check test is satisfied.

A-1.2.4 In the same manner 3-bromobenzanthrone and benzanthrone are purified.

A-1.3 Preparation of Column

A-1.3.1 Set up vertically a glass tube, length 40 cm and about 1·5 cm internal diameter joined with 50 ml thistle funnel at the upper end fitted with stop cock at the lower end. Place a cotton wool plug in the tube and press to the bottom of the tube by means of a glass rod, flattened at the end. Place a disc of filter paper. Cut to the approximate internal diameter of chromatographic tube on top of the cotton wool.

A-1.3.2 Prepare a slurry of about 50 g of alumina deactivated with 1 percent water in toluene and transfer it into the tube, wash down the side of the tube and pack the column by light tapping. Place disc of filter paper first and then a cotton wool plug at the top surface of the alumina. Always keep enough solvent in the column so that it remains wet and at least 2 cm of solvent layer shall always persist over the top of alumina. On no account allow the alumina to run dry. In the event of this happening re-slurry the alumina and repack.

A-1.4 Procedure

A-1.4.1 Weigh accurately about 60 mg of sample in 100-ml dry conical flask. Add about 50 ml of toluene and reflux for 10 to 15 minutes. Cool and transfer the solution to a 100-ml volumetric flask. Wash the conical flask well with toluene and transfer the washings to the flask. Dilute the solution to 100 ml mark.

A-1.4.2 Drain the excess solvent from the column so that the top remains just wet. Carefully transfer 100 ml of the sample solution with the help of a burette to the top of a chromatographic column. Add the solution to the column dropwise and at the same time drain the solvent from the column at same rate without allowing the top of the column to go dry. Fill the upper part of the tube with solvent and allow the chromatogram to develop keeping a good head of solvent above the alumina throughout.

A-1.4.3 Three bands will be separated out in column. Collect the first fraction when the band is about 1·5 cm from the lower tip of tube. This is a band of 3, 9-dibromobenzanthrone. When the band is eluted dilute it to 100 ml exactly. Then collect the second band of 3-bromobenzanthrone, in 250-ml volumetric measuring flask. After complete elution of same band dilute it to 250 ml mark with toluene.

A-1.4.4 Same way collect the third band which is of benzanthrone in 100-ml volumetric measuring flask with the completion of elution dilute it to 100 ml mark with toluene.

A-1.4.5 Adjust the wavelength of maximum absorption to the predetermined value and then adjust the instrument in such a manner that the transmittance through the blank becomes 100 percent after inserting the cell with blank solution.

Replace the cell with the solution of the sample and read the optical density.

A-1.4.6 Determine the optical density of the following:

a) 3, 9-Dibromobenzanthrone at	400 μm
b) 3-Bromobenzanthrone at	395 μm
c) Benzanthrone at	385 μm

A-1.5 Calculation

A-1.5.1 The assay of above fractions are calculated as follows:

$$\text{3, 9-Dibromobenzanthrone} = \frac{\text{Optical density}}{\text{mass in g} \times 0{\cdot}221\,8}$$

$$\text{3-Bromobenzanthrone} = \frac{\text{Optical density} \times 2{\cdot}5}{\text{mass in g} \times 0{\cdot}400\,3}$$

$$\text{Benzanthrone} = \frac{\text{Optical density}}{\text{mass in g} \times 0{\cdot}470\,1}$$

NOTE — Optical density of pure compound 1 mg per 100 ml will be affected to a certain extent by factors such as band width of the region (this depends upon the resolving power of the instrument) and accurate selection of wavelength. Therefore it is necessary to determine and verify from time to time the constants of the instrument that is being used.

A-2. DETERMINATION OF MELTING POINT

A-2.1 Determine the melting point of the sample in accordance with the method given under **8** of IS : 5299-1969*.

A-3. DETERMINATION OF MOISTURE CONTENT

A-3.1 Determine the moisture by drying in an oven at 100 to 110°C in accordance with the method given under **9.3** of IS : 5299-1969*.

A-4. DETERMINATION OF SULPHATED ASH

A-4.1 Determine the sulphated ash of the sample in accordance with the Method B given under **11** of IS : 5299-1969*.

A-5. DETERMINATION OF BROMINE CONTENT

A-5.1 Outline of the Method — 'IKA' Universal Bomb or Parr Bomb usually employed for the determination of halogens and sulphur in organic compounds, is used for fusion of the sample with sodium peroxide and ethylene glycol. The alkali bromide formed is determined by Volhard's method.

A-5.2 Reagents

A-5.2.1 *Ethylene Glycol*

A-5.2.2 *Sodium Peroxide* — granules.

A-5.2.3 *Standard Silver Nitrate Solution* — approximately 0·1 N.

A-5.2.4 *Standard Potassium Thiocyanate Solution* — approximately 0·1 N.

A-5.2.5 *Ferric Alum Indicator Solution* — 10 percent (*m/v*).

A-5.3 Procedure

A-5.3.1 Weigh exactly 0·2 to 0·3 g of the sample, by difference, in the tared fusion bomb. Add 7 to 8 drops of ethylene glycol, using a dropping bottle provided with a small pipette fixed in a rubber stopper. The tip of the pipette shall be so sized that 8 drops are equivalent to 160 to 170 mg of ethylene glycol. Fill up the bomb with granular sodium peroxide starting little by little to avoid sudden reaction. The bomb shall be filled up, tapping it to settle the contents, to 2 mm below the edge (about 11 g of sodium peroxide), care being taken so that no material is lodged between lid and the rubber gasket. After the bomb has been filled, replace the lid and *carefully close* the bomb manually. Place the closed bomb in the housing provided in the safety jacket plate. The tip of the microflame shall barely touch the bottom of the bomb.

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*Methods of sampling and test for dye intermediates.

A-5.3.1.1 Ignition should occur after 10 to 20 seconds (indicated by the red-hot colour at the bottom of the bomb). After a further 50 seconds, remove the bomb from the jacket with a pair of tongs and cool in a distilled water and open. The lid is thoroughly washed with distilled water and washings collected in a 500-ml beaker. Boil the fused mass along with the bomb in distilled water in the beaker covered with a watch-glass for half an hour, and allow to cool. Acidify the solution with concentrated nitric acid and cool again. To the cold solution add 25 ml of the standard silver nitrate solution and 15 to 20 ml of 10 percent ferric alum indicator solution till a reddish-brown end point is obtained. This back titration reading is noted as (*b*).

A-5.3.1.2 Perform a blank taking 25 ml of the standard silver nitrate solution and acidifying with concentrated nitric acid. Add 10 ml of 10 percent solution of ferric alum indicator and titrate against standard potassium thiocyanate solution till the same reddish-brown end point is obtained. Note this blank titre as (*B*).

A-5.3.2 *Calculation*

$$\text{Bromine, percent by mass} = \frac{(B - b) \times N \times 7{\cdot}992}{M}$$

where

N = normality of standard potassium thiocyanate solution; and

M = mass in g of the sample taken for the test.

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